

Synthetic Organic Chemicals

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Extraction Apparatus for the Laboratory

THE term extraction denotes the removal of definite constituents of a mixture by selective solution in some liquid in which they alone are soluble. Extraction falls into two classes, involving the treatment respectively of liquids and solids by liquid extraction media. The latter case will first be considered.

The classical method of doing this involves the use of a Soxhlet extraction apparatus, in which the condensed solvent flows onto the mixture of solids contained in a paper extraction thimble of special construction which serves both as container and filter, while the filtered solution is periodically transferred to the boiling-flask by means of an automatic siphon. This ingenious apparatus, in which the extraction is carried out at temperatures below the boiling point of the solvent, suffers from the disadvantage of permitting the extraction of only relatively small quantities, as the capacity of thimbles obtainable in the market is limited.

The extraction of quantities of solids up to about 500 g. may conveniently be carried out without the use of a Soxhlet apparatus merely by placing the substance in a fluted filter paper in an ordinary funnel stoppered to a flask containing the boiling solvent. The wide end of the funnel is loosely closed with a round flask, of larger diameter than the mouth

of the funnel, through which cold water is run (Fig. 1). The vapor of the boiling solvent condenses on the bottom of this flask, and the condensed liquid falls upon the material to be extracted; after percolating through this, it filters through the paper and runs down to the boiling flask. In this way the extraction is carried out at the boiling temperature of the solvent without the use of special apparatus and without particular attention. The water-cooled flask forms a surprisingly efficient condenser, and extractions may be performed even with ether without undue loss of solvent.

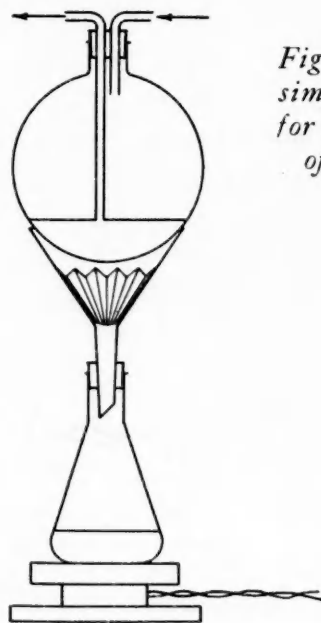


Figure 1 shows a simple apparatus for the extraction of solids up to 500g.

A different type of apparatus must be employed for larger quantities of solids. The material to be extracted may be

treated in a percolator; but, if this is not available, a satisfactory device may be constructed from a bottle of suitable size. This is filled to about three-quarters of its capacity with the solid to be treated, holding a wooden rod in position along the axis of the bottle during the process of filling. This rod is then carefully withdrawn so as to leave a channel, into which a tube is inserted. The apparatus, shown in Fig. 2, is then

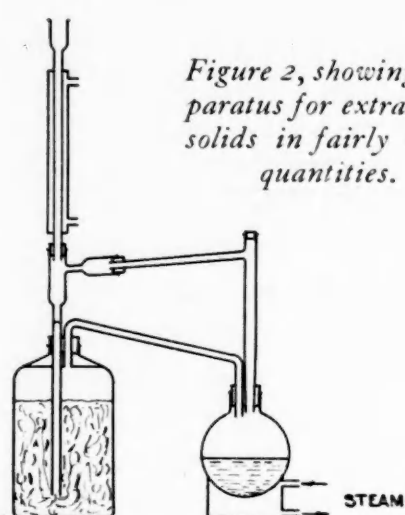


Figure 2, showing apparatus for extracting solids in fairly large quantities.

assembled and the bottle nearly filled with the extracting liquid, which must have a density appreciably lower than that of the solid. A second portion of the liquid is placed in the boiling flask, and gently boiled. The condensed liquid flows to the bottom of the mass and percolates upward, finally escaping from the exit tube and returning to the boiling flask charged with solute. Owing to the difference of the densities of the solid and the liquid, none of the former should rise with the upward stream of liquid to such an extent as to contaminate the out-flowing solution. The extraction may be performed at any desired temperature (below the boiling point of the solvent) by surrounding the extraction bottle with a suitable bath. This system is not highly efficient from a thermal standpoint, but has the advantage of requiring very little attention.

Extraction of substances in solution by immiscible solvents is usually effected in the laboratory by shaking in a separating funnel of capacity sufficient to accommodate the entire quantity. When volumes larger than one liter are to be handled, and only two or three changes of solvent are necessary, the shakings may satisfactorily be carried out in bottles; the liquids are separated by siphoning the bulk of the lower layer, decanting the major portion of the upper layer, and completing the separation in a small separating funnel.

When, however, more agitations are necessary to effect the extraction, continuous extraction permits great economy of time to be made. This operation is carried out exactly as in the extraction of larger quantities of solids in a bottle (Fig. 2), the latter being filled to rather more than three-quarters of its capacity with the solution to be treated. Here, again, it is imperative that the solvent should possess a lower specific gravity than the solution. This apparatus is readily adapted to the extraction of amino acids by Dakin's method with butyl alcohol, though it is advisable to modify it by the introduction of an extra condenser, preferably in the form of a water-cooled flask (Fig. 3).

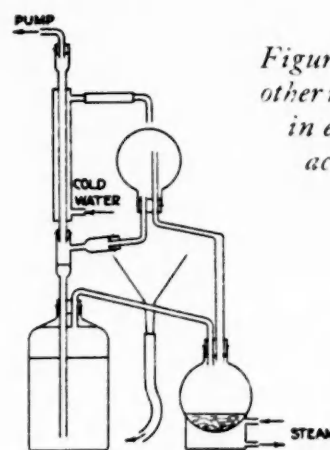


Figure 3, showing another modification useful in extracting amino acids by Dakin's method.

The extraction of solutions by liquids of higher density is less readily adaptable to continuous methods in the laboratory.

Organic Chemicals as Analytical Reagents

V REAGENTS FOR IRON

THIOGLYCOLLIC ACID

Lyons, J. A. C. S. 49, 1916 (1927)

Iron in solutions as dilute as one part in ten million can be detected by this reagent. To 5 cc. of the solution to be examined, which must be free of oxidizing agents, are added one drop of thioglycollic acid and 0.5 cc. of concentrated ammonia. A red or purple coloration shows the presence of iron. The color is due to a complex ferrous ion, any ferric iron present being reduced by the reagent. This reagent in conjunction with the thiocyanate method permits the colorimetric estimation of both ferrous and ferric iron in the same solution.

ALLOXANTIN

Deniges, C. R. 180, 519 (1925)

A blue color is produced by this reagent with iron salts in alkaline solution. The presence of citrates or tartrates which give trouble with the usual reagents for iron causes no difficulty in this case. The reagent is prepared by dissolving 0.1 gm. of alloxantin in 10 cc. of normal sodium hydroxide. Any color at this point should be destroyed by boiling and then cooling rapidly. Two cc. of a solution containing one milligram of iron per liter gives a distinct blue color with 1 cc. of the reagent.

DINITROSORCINOL

Nichols and Cooper, J. A. C. S. 47, 1268 (1925)

This reagent in neutral solution gives an olive-green color with iron, brown with copper, and orange-brown with cobalt. The limits of detection in milligrams per cc. are 0.0035, 0.0045, and 0.0033 in the order named. The reagent is prepared by adding an excess of the compound to boiling water and then filtering. Addition of a little sodium acetate increases the sensitivity to iron.

DIPHENYLAMINE

Knop, J. A. C. S. 46, 263 (1924)

One gram of diphenylamine in 100 cc. of concentrated sulfuric acid forms a suitable indicator in titrating ferrous salts with tenth-normal potassium dichromate. A mixture of sulfuric and phosphoric acids, also required, is made by mixing 150 cc. of sulfuric acid (s.g. 1.84) with 150 cc. of phosphoric acid (s.g. 1.7) and diluting with water to 1,000 cc. The ferrous solution is acidified with 20 cc. of 1:1 hydrochloric acid, 1.5 cc. of the phosphoric acid mixture, and 3 drops of the indicator. After diluting to 150-200 cc. the solution is titrated with the dichromate. The endpoint is taken as the last drop, causing an intense blue-violet coloration which remains unchanged after further addition of dichromate. A correction of 0.05 cc. is subtracted from the volume of dichromate to account for that used in oxidizing the diphenylamine.

New Chemicals

The following preparations have been added to our list since the last issue of this bulletin:

- **n*-Amyl Disulfide
 - Amylene (Tech.)
 - **n*-Amyl Iodide
 - **n*-Amyl Mercaptan
 - **n*-Amyl Sulfide
 - *3-Bromopentane
 - **p*-Bromophenacyl Bromide
 - **n*-Butyl Oleate
 - *Di-*n*-Amylamine
 - Ethyl "Oenanthate" (Tech.)
 - Hexaethylbenzene
 - **p*-Nitrosophenol Sodium Salt
 - *Pentane from Petroleum (Pract.)
 - o-Phenetidine (Pract.)
 - Phosphorus Pentasulfide (Pract.)
 - *Tri-*n*-amylamine
- *Made or purified in the Kodak Research Laboratories.*

A List of Inexpensive Organic Chemicals

AT the suggestion of the Division of Chemistry and Chemical Technology of the National Research Council, we have prepared the following list of inexpensive organic chemicals. They are listed roughly in the order of their prices.

The "Practical" or "Technical" grades of these chemicals are included in our price list to accommodate those who are interested only in small quantities for experimental work.

We shall gladly refer inquiries for large amounts to the manufacturers.

Benzene
Naphthalene
Cymene (Spruce Turpentine)
Dextrose (Glucose)
Toluene
Carbon Tetrachloride
Calcium Cyanamide
Chlorobenzene
Nitrobenzene
o-Nitrotoluene
Sodium p-Toluenesulfonate
Xylene
Ethylene Chloride
o-Dichlorobenzene
Ethyl Acetate
Formic Acid
Methyl Acetate
Methyl Alcohol
Oxalic Acid
Trichloroethylene
Formaldehyde (35-40%)
Tetrachloroethane
Ethyl Propionate
Acetic Acid
Acetone
Sodium Sulfanilate
o-Toluidine
Cresol (m and p mixture)
Acetyl o-Toluidine
Aniline
Dichloroethylene
m-Dinitrobenzene
Dinitrotoluene
Lactose (Milk Sugar)
Potassium Ethylxanthate
p-Dichlorobenzene
p-Toluenesulfochloride
Oleic Acid
Sodium Naphthalene- β -sulfonate
Benzotrichloride
Butyl Acetate
Chloroacetic Acid
o-Cresol
 β,β' -Dihydroxyethyl Ether
Maleic Acid
Stearic Acid
Urea
Tetrahydronaphthalene
Chloroform
Dinitronaphthalene
Ethyl Carbonate
p-Nitrochlorobenzene
Phenol
Tartaric Acid
Thiocarbanilide
Ethylene Glycol Monoethyl Ether
Furfural

Glycerol
Benzyl Chloride
 α -Chloronaphthalene
Ethylene Chlorohydrin (40%)
Paraldehyde
Butyl Alcohol
p-Dibromobenzene
2,4-Dinitrophenol
Tetrachloroethylene
Acetanilide
Acetic Anhydride
Calcium l-Malate (Sugar Sand)
Decahydronaphthalene
Diacetone Alcohol
Dimethylaniline
Methyl Ethyl Ketone
Methyl Sulfate
 β -Naphthol
 α -Naphthylamine
 α -Nitronaphthalene
Phthalic Anhydride
Propionic Acid
Salicylic Acid
Trichlorobenzene
Ethylene Glycol
Methyl Salicylate
Pentachloroethane
2,4-Dinitrochlorobenzene
Diphenylamine
Terpineol
Acetonitrile
Aldol
Amyl Acetate
Tert.-Amyl Alcohol
Amyl Phthalate
Betaine Hydrochloride
Butyl Propionate
Carbazole
Citric Acid
p,p'-Dichloroazoxybenzene
Ethyl Bromide
Indophenol (Paste)
Mucic Acid
p-Nitroacetanilide
o-Nitrochlorobenzene
Iso-Propyl Alcohol
Pyridine
Sodium 2-Chlorotoluene-4-sulfonate
Sodium Formaldehyde Sulfoxylate
Sodium p-Phenolsulfonate
Terpin Hydrate
p-Toluenesulfonamide
p-Toluenesulfonic Acid
Paraformaldehyde (Trioxymethylene)
Triphenyl Phosphate
Xylidine

Benzoic Acid
Cyclohexanol
Ethylene Bromide
Ethyl Phthalate
Hexachloroethane
dl-Malic Acid
m-Nitroaniline
p-Nitrotoluene
Safrole
Sodium Naphthionate
Tricresyl Phosphate
Acetyl Salicylic Acid
Anthracene (80-85%)
Benzaldehyde
Benzidine
Butyl Phthalate
Camphor
Chloral Hydrate
o-Chlorobenzoic Acid
o-Chlorophenol
Cyclohexane
2,5-Dichloronitrobenzene
2,4-Dichlorophenol
Diethylaniline
Diphenylguanidine
Ethyl Butyrate
Ethylene Glycol Monobutyl Ether
Ethyl Lactate
Ethyl Oxalate
Ethyl p-Toluenesulfonate
Gallic Acid
Lactic Acid
Methyl Benzoate
 β -Naphthylamine
p-Nitroaniline
Nitrobenzaldehyde (o and m mixture)
o-Nitrophenol
p-Nitrophenol
Phenyl Salicylate
Quercitrin (Lemon Flavine)
Sodium Picramate (Paste)
Sulfanilic Acid
Tannic Acid
Triphenylguanidine
Diphenyl Ether
Cineole
Ethyl Formate
Furfuramide
Hexachlorobenzene
Hexamethylenetetramine
p-Nitrophenetole
m-Phenylenediamine
Sodium Naphthalenedisulfonate
Tetrachlorobenzene
Triacetin